

# LABNOTES

Spring 1996

The Newsletter of the Wisconsin Laboratory Certification and Registration Program  
Program Info: (608) 267-7633 Program Fax: (608) 267-5231

## SPRING INTO CERTIFICATION RENEWAL

As the temperature rises and flowers blossom this spring, so will the laboratory business. A new season of sampling signals a new season of analysis, and most Wisconsin certified laboratories are required to renew their certification for the new fiscal year. Check your current certificate to see if your certification ending date is June 30, 1996. If it is, we would like to remind you that your lab must do several things prior to July 1, 1996 (December 31, 1996 for DMR labs) including:

- **Pass reference samples** for each test or test category (where required).
- **Pay the annual fee.** Bills will be mailed around the end of May and will coincide with any Environmental Fees Statements. Note that fees are scheduled to increase for next year to cover an additional certification officer.

Your laboratory will not be renewed for fiscal year 1997 until you have passed reference samples and paid the fees. If

you use the State of New York, APG, ASI or ERA reference samples for renewal, be sure to submit the results to our office. This is **YOUR** responsibility because we do not get copies directly from the providers. Results from the State Laboratory of Hygiene and the EPA are automatically uploaded into our database. Laboratories using the EPA WP and WS samples are encouraged to also analyze samples from one of the alternate providers because of uncertainty in the federal program. We cannot guarantee that the EPA WP035 and WS037 samples will be loaded in time for renewal.

### ***Review your Certificate!***

After paying the renewal fees and submitting acceptable reference samples, your laboratory will receive a new FY 1997 certificate and an exception report in early July. Carefully read the certificate and exception report and double check to make sure that it is correct. It is much easier to solve certificate misprints early in the fiscal year. The exception report will detail which parameters didn't print on your certificate and why.

After reviewing the certificate and exception report you should contact us if you do not

(Continued on p. 2)

### **In this issue...**

<b>Fees to Increase</b> .....	<b>2</b>
<b>Lab Cert Rules Effective</b> .....	<b>2</b>
<b>SLOH Announces PE Testing Policy</b> .....	<b>3</b>
<b>Compound Removed from Landfill List</b> .....	<b>4</b>
<b>New WET Testing Requirements</b> .....	<b>4</b>
<b>Laboratory Certification in the Reorganized DNR</b> .....	<b>5</b>
<b>Who is in Charge of Microbiological Testing</b> .....	<b>6</b>
<b>List of Compounds with Standards Near the LOQ</b> .....	<b>6</b>
<b>Auditor's Corner by Alfredo Sotomayor</b> .....	<b>6</b>
<b>Leak Testing in ECD's</b> .....	<b>11</b>
<b>DNR News Briefs</b> .....	<b>11</b>

## SPRING INTO CERTIFICATION RENEWAL

(Continued from p. 1)

understand why the certificate does not reflect your expectations.

### ***Reciprocity Certification:***

Reciprocity labs need to send updated copies of their host state's certification or accreditation and pay the reciprocity fee before we can renew the reciprocity certificate. It is your

responsibility to get the certificate to our office.

***Deadlines:*** The deadline for renewal is June 30, 1996. Failure to pay the annual fee prior to June 30, 1996 will result in an additional late fee penalty to be assessed to your laboratory. If you still have not submitted acceptable reference samples or have not paid your fee by September 1, 1996 your certification or

registration will **EXPIRE** and you will need to submit a new application to enter back into the program. It is very important to meet the necessary deadlines to help keep the program running efficiently and consistently. Renewal went very smoothly last year, and we expect a repeat performance this year. Questions on your renewal should be addressed to John Condron at (608) 267-2300.

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## FEES TO INCREASE FOR FISCAL YEAR 1997

The Department has received authorization from the Natural Resources Board at its March meeting to increase the fees for certification and registration according to the new fee formula which is found in Ch. NR 149. Using the formula, the cost per relative value unit will be \$35. The increased fees will generate an estimated \$87,900 for the program which will be used in part to fund one additional certification officer.

Renewal bills for certification will be mailed to all certified and registered laboratories in late May 1996 and will be due June 30, 1996. Labs which do not pay their bills prior to the deadline will be assessed a late payment fee. The absolute deadline for payment of the bill plus the late fee is September 1, 1996. Questions about your bill should be addressed to John Condron at (608) 267-2300. The fees for each category are as follows:

Program Base Fee -	\$350.00
Cats. 01 - 04-	\$35.00
Cats. 05 - 06-	\$70.00
Cats. 07 - 14, 16, 19	\$140.00
Cats. 15, 17-	\$420.00
Cat. 18-	\$700.00
Cat. 20-	\$910.00
Minimum Fee-	\$840.00
Reciprocity Fee-	\$840.00
Initial App. Fee-	\$210.00
Revised App. Fee-	\$105.00
Late Payment Fee-	\$70.00

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## NEW CERTIFICATION RULES AND METHODS TAKE EFFECT

The amendments to Chs. NR 149, 219 and 700 approved by the Natural Resources Board in September of 1995 became effective March 1, 1996. Copies of the new rules are being distributed through the state's







subscription service, and many of you may have already received copies. The Laboratory Certification Program intends to distribute copies to all certified and registered laboratories when

the program's "Yellow Book" is updated this spring. Some of the highlights of the new rules include:

(Continued on p. 3)

## NEW CERTIFICATION RULES, METHODOLOGIES TAKE EFFECT

(Continued from p. 2)

 Including the SW-846 methods in Ch. NR 219 for wastewater.	 Implementing a formula for calculating fees for the program.	Many other changes were made as well. The Department intends to hold public informational meetings on these rules on April 31 and May 1 of this year to answer questions about the rules. See page 12 for more information. Questions or comments about the new rules should be addressed to Jeff Ripp at (608) 267-0579.
 Requiring methanol preservation for all GRO and PVOC soil samples.	 Changes to test category structure for semivolatiles and pesticides.	
 Promulgating the September 1995 GRO and DRO methods as authoritative sources in Ch. NR 149.	 Requiring laboratories to report down to their limit of detection for many substances.	

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## STATE LABORATORY OF HYGIENE ANNOUNCES POLICY FOR ENVIRONMENTAL PROFICIENCY TESTING PROGRAM

The Wisconsin State Laboratory of Hygiene (SLOH) Environmental Proficiency Testing Program has made many, many changes over the last year to try to serve both the labs and the DNR better. They continue to make improvements in their program and have enacted several policies regarding the reference sample program. Be advised that these ground rules will apply to **all laboratories**, and no exceptions will be made. Questions, comments or concerns about the Environmental Proficiency Testing Program should be addressed to Barb Burmeister at (800) 462-5261, ext. 107. The fax number for the proficiency testing program is (608) 833-2803.

### ***Replacement Sample Policy:***

Replacement sample requests must be made within 10 days from the ship date. It is the lab's responsibility to examine the shipment as soon as it arrives and to report any missing or damaged samples to the SLOH. Replacement requests due to mishandling of samples (misdilution, spillage, etc.) on the part of the participant must also be made within the 10 day period. In order to meet the replacement request deadline, the SLOH encourages labs to begin sample testing as soon as the shipment arrives. A fee will be charged for samples that have been mishandled by the participant. This fee will cover the cost of additional samples, shipping and handling charges.

### ***Additional Sample Policy:***

Samples for uses other than reporting proficiency testing results (e.g. evaluating new methods or instrumentation, trouble shooting proficiency testing failures, staff training) may be purchased as supplies are available. The price will include the cost of the samples, shipping charges and handling fee.

### ***Extension Request Policy:***

In case of temporary equipment problems, an extension of the data form due date may be granted at the discretion of the program coordinator for up to two weeks from the original due date. Requests for extensions must be received prior to the deadline.

(Continued on p. 4)

## STATE LABORATORY OF HYGIENE ANNOUNCES POLICY FOR ENVIRONMENTAL PROFICIENCY TESTING PROGRAM

(Continued from p. 3)

Requests for extension received after the deadline will not be considered.

### ***Late Results Policy:***

Proficiency testing results (data forms) must be postmarked by the Post Office on or before the due date appearing on the data form, unless an extension has been granted. Alternately, results may be submitted via FAX (608-833-2803) on or before the due date. Results

postmarked or FAXed after that date will not be graded, and the lab will receive a score of "NR (no results)" on their report and the data will not be usable for certification in Wisconsin.

***Participation Policy:*** If the WSLH did not receive results for a sample for which a participant enrolled, the participant will automatically receive and be billed for that sample in subsequent PT event shipments. If it is determined

that a sample or analyte can not be graded, the participant will also automatically receive and be billed for that sample in the subsequent event.

***Sample Failure Policy:*** If a participant receives a score of <80% (failure status) for an analyte in a sample that contains more than one analyte (e.g. organics), the participant must repeat the entire sample in the next testing event and not just the failed analyte.

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## 2-CHLOROETHYL VINYL ETHER REMOVED FROM SUBTITLE D MONITORING LIST

vinyl ether is listed on form 4400-107A, the DNR's Solid Waste Program has decided that it does not need to be reported on the form. This compound does not appear on the Subtitle D (federal Landfill Rules) monitoring list. However, this parameter is regulated differently under different programs and is still listed in methods 601, 624, 8010, 8240, and 8260A (yellow pages). It is not listed in 8021 or 8260. This

wastewater priority pollutants and the EPA Hazardous Constituents List (40CFR264 Appendix VIII). It is not listed in ch. NR 140, Wis. Adm. Code. This compound was deleted from 40CFR257 & 258 Appendix I in the 10/09/91 Federal Register page 51075.

According to the Merck Index, 2-chloroethyl vinyl ether will break down into acetaldehyde and 2-chloroethanol in dilute

sodium hydroxide solution. Samples for this analyte should not be preserved with acid. Laboratories wishing to analyze for 2-chloroethyl vinyl ether may want to consider preserving the sample in a dilute sodium hydroxide solution to ensure sample integrity. For more information, contact Janet Battista in the DNR's Bureau of Solid and Hazardous Waste at (608) 267-3533.

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## NEW REQUIREMENTS FOR WHOLE EFFLUENT TOXICITY TESTING

The process of codifying Wisconsin's requirements for conducting whole effluent toxicity (WET) tests is in its final stages. The State of

Wisconsin Aquatic Life Toxicity Testing Methods Manual is the result of efforts in the Department to streamline the permit issuance

process and to update all current WET testing requirements found in the

(Continued on p. 5)

## NEW REQUIREMENTS FOR WHOLE EFFLUENT TOXICITY TESTING

(Continued from p. 4)

"Guidance Manual for the Certification and Registration of Laboratories Conducting Effluent Toxicity Testing." After codification of the Methods Manual, permittees and laboratories will no longer have to refer to four separate documents when looking for Wisconsin WET testing requirements. The most significant changes to the WET testing requirements include:

- ★ A holding time of 36 hours will replace the current one of 48 hours.
- ★ Samples must arrive at the laboratory at 10 °C or less. If samples do not meet these temperature requirements they must not be analyzed except when the sample arrives at the laboratory within 4 hours of collection.
- These samples may be analyzed.
- ★ Daphnia Magna will no longer be used in WET tests.
- ★ The Methods Manual contains a table which specifies ranges of hardness which are easily obtainable using standard USEPA recipes.
- ★ Whenever ammonia is present in the effluent at a level which may cause artifactual ammonia toxicity to confound WET test results, the tests must be conducted in a 2.5% CO<sup>2</sup> atmosphere.
- ★ For acute tests, the age of fathead minnows is 1 to 14 days old instead of 20 to 60, the current requirement.
- ★ Acute WET tests will be done using a series of effluent treatments and an LC50 must be generated in order to estimate the lethality of an effluent.
- ★ The Methods Manual requires that a four page report be filled out and submitted to the WDNR. This significantly reduces the amount of information that must be submitted to the Department.

The test procedures found in this Manual are not more stringent than those promulgated by the USEPA, but instead are written specifically for situations found in Wisconsin and are more appropriate for the DNR's WET testing program. Contact Debra Piper at (608) 264-8950 for more information about the program or for information regarding WET testing certification.

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## LABORATORY CERTIFICATION IN THE REORGANIZED DNR

The Laboratory Certification Program is moving to a new Division and Bureau through the reorganization of the agency. The program will be part of the Division of Enforcement and Science. It will reside in the newly created Bureau of Integrated Science Services. This Bureau was created by the consolidation of the Office of Technical

Services and the Bureau of Research and Environmental Review. Lab Certification will remain with the Analytical Services Section, one of nine sections in the new bureau.

Movement of this program in the agency will not cause any interruption in service to the 575 labs currently registered or certified. Also, no delays in the

renewal of certifications or registrations is expected. The location of the office itself and phone numbers will not change. If you would like more information regarding the reorganization of the DNR contact Jack Sullivan at (608)-267-9753.

## WHO IS IN CHARGE OF MICROBIOLOGICAL TESTING CERTIFICATION?

We have received several questions of late regarding microbiological certification and we would like to clarify that microbiological testing (e.g. coliform testing) for drinking water and milk is not the responsibility of the Department of Natural Resources (DNR). In past years the Department of Health and Social Services

(HSS) has had the responsibility for milk and water microbiological certification. However, in the 1996-97 biennial budget bill the responsibility was transferred to the Department of Agriculture, Trade and Consumer Protection (DATCP). DATCP is currently writing rules for the program with input from DNR

Water Supply Staff. The staffing (which will remain the same) is being transferred from HSS to DATCP. Laura Forst is the certification officer for the program and she is located in Green Bay. If you have any questions on program requirements or applications, contact Laura at (414) 448-5346.

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## COMPOUNDS WITH STANDARDS NEAR OR BELOW THE LOQ

The Department is required to annually publish a list of compounds with standards that are at or below the limit of quantitation as defined in NR 149. Likewise, NR149 requires laboratories to report results down to their detection

limit for any compounds that have standards below the limit of quantitation. However, many programs require the reporting of all results above the limit of detection regardless of the standards. Be sure to check the with your

clients to determine their low-level data needs and the Department's requirements. The compounds of potential concern across all agency programs are listed below:

1,1,2,2-Tetrachloroethane

1,1,2-Trichloroethane

1,1-Dichloroethylene

1,2-Dibromo-3-chloropropane (DBCP)

1,2-Dibromoethane (EDB)

1,2-Dichloroethane

1,2-Dichloropropane

1,3-Dichloropropene (cis/trans)

2,4-Dichlorophenoxyacetic Acid (2,4 D)

2,4-Dinitrotoluene

2,6-Dinitrotoluene

Alachlor

Aldicarb

Aldrin

Antimony

Arsenic

Atrazine, total chlorinated residue

Benzene

Benzidine

Benzo(a)pyrene

Beryllium

Bis(chloromethyl)ether

Bromodichloromethane

Bromoform

Bromomethane

Butylate

Cadmium

Carbofuran

Carbon Tetrachloride

Chlordane

Chlorine (total residual)

Chloroform

Chloromethane

Chromium

Chromium (Hexavalent)

Cyanazine

Cyanide

DDT and Metabolites

Di(2-ethylhexyl)phthalate

Dibromochloromethane

Dimethoate

Dinoseb

Dioxin (2,3,7,8-TCDD)

Endosulfan

Endrin

Formaldehyde

Heptachlor

Heptachlor epoxide

Hexachlorobenzene

Lead

Lindane

Mercury

Methoxychlor

Methyl ethyl ketone (MEK)

Methyl isobutyl ketone

(MIBK)

Methyl tert-butyl ether

(MTBE)

Methylene Chloride

(Dichloromethane)

Metolachlor

N-Nitrosodiethylamine

N-Nitrosodimethylamine

Naphthalene

Parathion

Pentachlorophenol

(PCP)Polychlorinated

biphenyls (PCBs)

Selenium

Simazine

Silvex (2,4,5-TP)

Tetrachloroethylene

Tetrahydrofuran

Thallium

Toxaphene

Trichloroethylene (TCE)

Trifluralin

Vinyl Chloride

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## THE AUDITOR'S CORNER

Alfredo Sotomayor, Senior Audit Chemist

### YOUKALI LABORATORIES, LTD.

When I called to make the appointment, Ms. Brown said she was looking forward to my visit, but before I could ask her to send me the floor plan and the latest copy of the Quality Assurance Manual, she added:

"I want you to really see how we do things here. We are not certified by many states, or A2LA, and we are not a CLP lab, but we do things right. We do them differently, but we do them right."

(Continued on p. 7)

### THE AUDITOR'S CORNER

(Continued from p. 6)

"How differently?" I ventured, bracing myself for yet another set of deviations.

"Sometimes, quite differently," she responded. "But let's not get into that right now. When you get here, you will see what I mean."

"I'd rather not deal with surprises," I said emphatically. "Can you send me a floor plan, a list of analytical equipment, a personnel roster, the latest copy of your Quality Assurance Manual, and written directions to your laboratory?"

"Of course, Mr. Sotomayor. Is there anything else that you may need to see before your visit?"

"No, just make sure the directions are good."

"Very well. I am looking forward to meeting you; I have heard many things about you."

"Yes, word gets around," I said. "I hope the word that's getting around these days is charming, if not fair."

"A little of both," Ms. Brown replied, almost suppressing a chuckle.

"Goodbye, Ms. Brown." I ended feeling like Rodney Dangerfield. But who's to blame?

Two days before the audit, I received a package containing the information I requested. The Quality Assurance Manual was thin, and in my business, brevity is bliss. To my astonishment, the Manual dispensed with all the usual formalities--no Policy, Mission, or Vision statements. Instead, it contained seven sections, but each referred me back to documents "controlled at the facility". I was beginning to fear that Youkali was devoted to evasion, or at best, delayed customer gratification.

And then, getting there was not a pleasant experience: a long drive from Madison, a hotel in the middle of nowhere, and in the morning, following an intricate, but correct, set of directions, feeling I was going to the end of the world, until I saw the sign in gleaming silver against a white background: **YOUKALI LABORATORIES, LTD.** There also was the curious logo: planet Earth, covered with clouds, except for a tiny silver star, radiating light into the letters forming the lab's name. It looked more distinctive on-site than on the letterhead.

The building was small. The visitor's parking lot had a sign saying: Welcome to Youkali! "Indeed," I thought as I picked-up my briefcase slightly dizzy from having to negotiate too many turns. Oh, one other thing made me pause: the building was immaculately white. The door hinges, the window frames, the extractor towers were all white! I left my sunglasses on.

Inside the lobby, another sign, with my name correctly spelled, welcomed me once again to Youkali. I started to hum "Hotel California", and stopped startled after a few bars. I hoped there would be no connection, when without being paged, a woman, dressed--you guessed--all in white, approached me.

"Good morning, Mr. Sotomayor. I am Lucille Brown."

"Good day, Ms. Brown. How do you do?"

"Lucy, please. Would you like some coffee?"

"Alfredo, please. Coffee is just what I need. Thank you, Lucy."

"Please follow me. I have reserved the conference room for the whole of your stay."

And so the preliminaries went. After a few pleasantries, I was introduced to seven other people, mercifully not in white. However, each was wearing only a single primary color. Trying to associate a face with a name, I wrote: the woman in red, the man in blue, the man in green, the woman in yellow...

I began, "I would like to organize the tour by playing 'sample' and starting from the point of receipt, go throughout the entire lab."

"Oh no. Not here," Lucy politely remarked. "Why play that boring game when you could do things differently and get a good sense of what is unique about us? I have set the most interesting agenda for you..."

"Lucy, I am the auditor. I must be allowed to set the schedule and..."

At this point, the man in blue, who my notes told me was the laboratory director, interrupted: "Lucy is right. Why bore yourself with such foolishness. It is not very useful here, trust me."

Now I could not get that Eagles song out of my head: "Such a lovely place..."

"Excuse me. We do things differently in my program."

"Oh we know," said Lucy. "That is why we think you'd enjoy trying it this way."

"And exactly what is your way?" I asked, perceptibly annoyed.

"Let me give you an overview of our Quality Assurance Program."

Lucy and the rest of the primary-color-attired people remained silent, looking at me expectantly. I considered my options and just when I thought I would deliver a terse admonition, I heard myself, as if possessed, saying:

"Very well. I will give you thirty minutes to complete an overview. After that, we will proceed with the tour."

(Continued on p. 8)

## **THE AUDITOR'S CORNER**

(Continued from p. 7)

Lucy beamed a radiant smile. The rest, in unison, turned their heads to me. A tango, or something like an habanera, was now softly audible in the room, but I was still hearing "Welcome to the Hotel California...Such a lovely place..."

"Alfredo," Lucy commenced, "the cornerstone of our Program is that we distinguish between what is achievable under ideal and daily circumstances, what is achievable for each sample, and what can be extrapolated for a set of samples. We have an elaborate flagging system that is attached to each datum produced by Youkali. We like to think of it as all our numbers having documented pedigrees. We know that some numbers are better than others. We do not even pretend that all results are of equal validity, but here, you can determine the validity of each result by following a simple key.

"And how is this different from the CLP flagging?," I asked, fearing that giving her thirty minutes had been a mistake.

"Good question. We do have minimum criteria that exceed those of the CLP because we have done away with one fallacy."

"And what is that?," I asked, this time, curiously.

"We do not let multi-laboratory performance dictate our minimum requirements. If the data used for multi-laboratory acceptance limits were derived from the best laboratories, then I would use it to judge performance, but you and I know that this is not the case. Let me ask you, if you wanted to go to Harvard or MIT, would you compare your grades to those from a national average, or would you look at those from a set percentile? Doesn't it make sense then to be selective in choosing these criteria?"

"Yes, but that presumes that everybody wants to or needs to get into Harvard or MIT," I responded, interested in what Lucy would now offer.

"Correct. Let's deal with 'wanting' to first. Given a chance to market its quality, a laboratory would try first to compare itself to Harvard than to the 'Acme International Psychic University' of environmental analysis. When it comes to quality, the best is the ultimate goal, even if not always achievable."

"And how about those that do not need to have results from an Ivy League Laboratory?"

"Well, that one is not as easy to answer," Lucy replied, "but even clients not needing such a high-quality of service would benefit from the superior treatment." Lucy added, as if anticipating my remark, "Usually, cost is the reason why some feel that quality of service should be commensurate with the payment received. Although at times you do not need a Cadillac when a Volkswagen will do, wouldn't you expect that the Volkswagen be perfectly tuned, safe, and cute as a bug. I am sorry."

The primary colors did not change hue. Lucy continued unperturbed, "The point I want to make is that each level

of service should have the best quality as its ultimate goal. Anyhow, here at Youkali we found that the associated increased costs were, although real, not as significant as others believed. We also discovered that by treating samples uniformly, instead of segregating them into various tiers of quality requirements, we saved some money by eliminating additional monitoring systems and avoiding embarrassing reanalyses."

"So what does this mean, in practical terms? Do you offer extensive narratives and thick packages with all your results?," I asked, a little confused. "Your Quality Assurance Manual was a marvel of brevity and lightness."

"Oh, no, on the contrary. Our reports are not extensive because we do not have to justify as much as others operating under laxer systems. Our Quality Assurance Manual reflects our policy of delivering exactly what is needed. What you got was exactly that--what you needed at that time."

"Frankly, what you sent me left me thinking you were not telling me the whole story."

"Exactly, Alfredo. That is why I wanted you to start the audit in this manner."

"OK. Can you give me some concrete examples of your pedigreed approached to analytical services?"

"I was talking about Harvard and MIT before and the analogy was appropriate. We treat analyses as if they were going to school. All results reported by Youkali receive grades. Projects do not graduate until they have passed certain courses."

"Really? Sort of like Calibration 101, Surrogate 507, or Advanced Spiking?," I asked, envisioning a VOC vial with a teflon-lined mortarboard, and the man in blue, this time in an indigo academic gown, giving the vial a report certifying, in Latin, that its results were ready to be released.

Lucy smiled politely. "Almost, you will see. But my half-hour is over, what would you like to do now?"

I knew then that Youkali was going to be unlike any other lab I had audited. And so I said, "Please Lucy, continue with your presentation."

As if on cue, Lucy pulled a screen showing a circle with the word 'calibration' in it. She continued, "Let's look at the fundamentals. We start by assuring that all initial calibrations pass criteria for all analytes. We do not move forward unless an initial calibration has been performed properly."

"Does that mean, for instance, that for organic methods by GC/MS, you look at the CCCs and the SPCCs, and if they meet method criteria, the calibration passes?"

(Continued on p. 9)

## **THE AUDITOR'S CORNER**

(Continued from p. 8)



"No. We look at the SPCCs for minimum response, but that is almost always met, so it is rarely indicative of the quality of a calibration. We look at ALL compounds, and make sure all of them meet the strictest criteria established in a method of similar ilk. We use the strictest, because this give us longer-lasting calibrations. Without getting into calibration algorithms, let's just concentrate on using selected compounds to evaluate a whole GC/MS calibration. That is for the dodos."

Lucy noted I looked puzzled, "You know, for the birds. It is not done for GC work and some regulatory programs, like the SDWA, have never allowed it. I think those that advocate this selective practice want to extrapolate the unmistakable identification power of the GC/MS to quantitations, and that is not justified. Just because you can see it, does not mean you will quantitate it correctly. Anyway, how can one justify controlling a compound strictly, and then allowing a very similar one to be all over the place?"

I agreed, but fell compelled to raise the argument I had heard so many times: "But doesn't it follow that if you have to analyze so many compounds, you can miss some, just by chance, not because of a system failure?"

"Yes, who can deny that. But you know, I have not found a good way of determining when an analyte fails, that such a failure is the result of a statistical inevitability. I think we would like to assume that, but the affected compound could be quantitated inaccurately, whether the failure be related to the system or to chance. Have you ever heard anybody claiming that an initial calibration met criteria for all compounds simply by chance? No, successes are usually attributed to design, while failures...well. Chance failures should not be repeatable. Reanalyzing the standards for the affected analyte could detect a pattern hinting at a system failure. But, what can happen is that while a failing compound passes on reanalysis, one that had previously passed, now fails. The problem here is trying to analyze too many compounds under the same conditions. One size cannot possible fit so many."

"And what is one to do?," I asked hoping she could give me some radical ammunition.

"Make analyses come in different sizes," she responded almost triumphantly.

Oh no, from academics to couture, I thought. How will I ever explain this to my boss?

She continued. "Sometimes it is necessary to customize analyses for certain compounds. And if you follow the 'so many compounds, so many more failures' argument, then analyzing fewer compounds at a time should lessen the possibility of failing one just by chance. At Youkali we started by trimming our multi-analyte lists to the essentials. We only give you what you need. We also

have accepted that some compounds fall more rapidly out of calibration than others. For those, we have optimized chromatographic conditions, and run them separately."

"But that increases costs. How can you afford to be in business?," I asked.

"Yes, it costs more. But the clients we have value our customized services. They come to us seeking excellence, not mediocrity. We treat their environmental samples as if they were blood samples from loved ones. But, I am getting a little carried away. To return to my presentation, this means that since no result is reported before an initial calibration passes, there are no flags in this laboratory for initial calibration failures."

"Are there flags for continued calibration verification failures?," I inquired.

With characteristic emphasis, Lucy responded, "No. All CCV failures are solved before analyses proceed. All reported results are associated with an A or a B for CCV performance."

"You really grade them, don't you? What gets an A?"

"All CCV checks within 10% of an analyte's original response or actual concentration get an A. Those outside 10% , but within 20% , get a B. A grade of C in this 'class' is not acceptable for graduation. If a method allows more than a 20% acceptability window, we do not. Most inorganic procedures require an A in this class."

I was beginning to see what Lucy meant and my mind was racing; what about laboratory control samples, spikes, duplicates. The GPA for sample 3266? A summa cum laude report for sample 7745?

Lucy pulled a second screen with a bigger circle and the word 'laboratory control sample' in it. The primary

Lucy pulled a second screen with a bigger circle and the word 'laboratory control sample' in it. The primary colors momentarily turned their gaze from me to give Lucy a visual nod to proceed.

"We also grade the results of LCSs by a similar scale. Here though, we allow up to a C, a recovery outside of a 20%, but within a 30% window, as acceptable. A grade of D or F make a batch fail this class."

Lucy pulled another screen graced with a circle. "For those methods amenable to surrogates, we grade their

(Continued on p. 10)

## **THE AUDITOR'S CORNER**

(Continued from p. 9)

recoveries similarly, giving a D to recoveries outside a 30%, but within a 40% window. Recoveries outside the 40% window get Fs."

"So a sample can fail a surrogate and still graduate?," I asked, disillusioned.

"Yes, unfortunately. But, an initial grade of D or F for a surrogate requires investigation and possibly reanalysis. If after this, the surrogate still has a D or F, then, if the rest of the sample's grades are good, the sample graduates."

"What about analyses for which surrogates do not exist? What about metals?"

"Well, those samples do not have to take that class, if you follow. Different majors require different courses," Lucy responded. "But since you asked for metals analyses, for our ICP we now use yttrium as an internal standard, and so finding a surrogate cannot be too far away. AA will be dead early in the next century. But here I go again, making categorical pronouncements."

I was now humming along the infectious tango that the room's speaker delivered. I noticed that Lucy's presentation moved along with the rhythm of the tune. No wonder it all seemed to fit.

"So when does a sample graduate?," I asked, curbing a sudden impulse to get up and dance to the nice tango beat.

"Well, not yet. We still have to deal with matrix QC."

"Oh," I said, a little less elated.

"We have done away with computing matrix control limits statistically," Lucy continued. "We felt that the underlying principle behind these calculations was not always operant in a laboratory analyzing environmental samples from various sources. Charts and the Shewhart approach to control makes sense when a population is well-defined. For instance, determining the 'three sigma' control limit for measuring a characteristic of a manufacturing process, say, the tensile strength of a copper wire, makes sense. Getting a result outside of this interval would indicate that there was a significant difference in the manufacturing process."

"However," Lucy explained seamlessly, "environmental samples are not all one big, happy family. Making inferences and reaching conclusions about a set of soils from a single spiked sample may not be very defensible. It seems then that to demonstrate control at the matrix level, you should spike and duplicate each sample in a batch. Well, even at Youkali, that is not feasible. And since we do not have analogs for each compound we determine, spiking each sample would be extremely complicated."

"So what do you do? Do you not analyze matrix spikes and replicates?"

Down came screens with the words 'matrix spikes' and 'replicates'. "No, we analyze them," Lucy answered. "However, we grade recoveries on the same scale we use for surrogates. Replicates or matrix spike duplicates are graded similarly, using RPD. Results within 10% RPD get an A, results higher than 40% RPD get an F. We investigate all recoveries and RPDs with F grades. Investigation means looking at background concentrations, reanalyzing extracts or digestates, and checking for process anomalies. If a satisfactory explanation is not found, then re-processing of the spiked sample proceeds if there is enough sample. The results of the re-analyses are then reported with their grade. If re-processing of the original sample spiked is not possible, then another one in the batch is reprocessed. When this is also not possible, the batch gets an F."

"Isn't this penalizing a whole batch for the failure of one single sample?," I asked in spite of myself.

"Yes; life is unfair sometimes. But, this forces us to select an adequate sample for spiking, pay attention to the concentrations we use for spiking, and use clean-up techniques more frequently. A spike and a replicate are also judged on their own merits: associated LCS and surrogate recovery, calibration verification, etc. All of this is evaluated before graduating a sample or a batch."

"So it is possible for a batch to get an F for spike recovery and still graduate if the rest of the course work is sufficiently excellent?," I added.

"Precisely. The batch won't graduate with distinction, but it would still graduate."

"You know Lucy, there is something that's been bothering me that your presentation hasn't addressed. How do you deal with methods that are, to say it politely, not rugged? How do you deal with a method that allows a recovery for a compound from detection to 199%?"

"Good, I was waiting for that question," Lucy replied with great zest. "Are you referring perhaps to a well known HPLC method?"

"Perhaps, and others" I added, nodding emphatically.

"Well," she continued, "remember that we do not look at broadened intra-laboratory performance data for establishing our acceptance limits. But, there are some methods not up to our standards that we are forced to use. In these cases, we designate the methods as 'Pass or Fail', just like that tennis course you took in college."

"I took yoga."

"And I took fencing," Lucy added laughing. "For those methods, we do not grade LCSs, surrogates, matrix

(Continued on p. 11)

## **THE AUDITOR'S CORNER**

(Continued from p. 10)

spikes, and replicates by our strict scale. Recoveries between 50 and 150% get a P for passing, as do RPDs equal or less than 50%. Failures are investigated as we already discussed. Historically low LCSs recoveries tell us that a method should be taken only as a Pass or Fail for 'credit'. We know about this before we analyze samples because we always do IDCs. Also, no more than two non-passes are allowed, if you want a sample to graduate, and, we never compromise with calibrations. Continuing calibrations are still strictly graded, as I have already explained."

"So what's left? How do you get a diploma?," I asked.

"Ta-ran! To graduate, all analytes reported for a sample must have an average grade of at least C, or a P, for pass, for those few problematic methods. In the end we tally all the analyte results and give the sample its final grade. When the client gets results, the client will see all grades and the final grand average, all of which will telegraph the quality of our work."

As Lucy concluded, I noted that all the screen circles, when superimposed formed a bulls-eye pattern. Neat, I thought; this explains the interconnectedness of quality. The primary colors now stood up, held hands, and circled Lucy as they all moved to the tango beat.

"Great presentation. Thanks!," I remarked as I also got up and did a quick one, two step. "I am ready for the laboratory tour."

Curiously, now I overheard a faint siren over the tango tune. Lucy and the colors continued to twirl and smile and I was getting a little dizzy, as they continued to move faster and faster. Now it seemed the colors were all blending into the purest white. "I better get-on with this tour fast," was my only thought. The siren got louder and louder, finally drowning the tango. I realized a tornado must have been sighted and there was no time to lose. Just as I was preparing to seek shelter, the room shook and the colors and Lucy seemed to fuse into something quite like Youkali's logo. The siren now was piercingly loud...I was looking for Toto, even though I knew I had arrived unaccompanied.

"This is NPR," I heard above the din, dreading the impending catastrophe. "National Public Radio. This is the Morning Edition of *All Things Considered*."

"Oh goodness! It was just a dream," I exhaled relieved. The siren had been my double alarm; I need all the help I can get to wake-up. Thinking again about this most bizarre vision, I now felt saddened that it was just a dream and before getting slowly up, hummed, to the nice tango tune still in my head: "But it's just a dream, it's just folly. There is no place like Youkali."

*Ed. The name of this fictional laboratory is traceable to a real source. Give yourself a pat in the back if you can identify it. Let us know if you discover it.*

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## LEAK TESTING FOR <sup>63</sup>Ni IN ELECTRON CAPTURE DETECTORS

One question that has been raised frequently relates to certification requirements necessary to retain instruments on site which contain small quantities of radioactive materials. One example of this is the <sup>63</sup>Ni foil used in electron capture detectors. We have learned that laboratories can be covered under the umbrella of the license held by their instrument manufacturer. These

laboratories are still required to perform leak testing every 6 months to meet the requirements of 10 CFR Part 31.5 (c) (2). Your service or sales representative may be able to coordinate this testing through the instrument manufacturer, but other contractors are available as well. At this time we have identified one local firm that performs leak testing of these instruments for laboratories.

Most current cost information suggests approximately \$28 per instrument, but quantity discounts may be available. For more information, contact Wisconsin Radiological Laboratories, 3802 Packers Avenue, Madison, WI 53704, (608) 244-4646, (800) 773-7731 (in WI only), FAX: (608) 249-8098.

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## NEWS BRIEFS

**1) The Update to Program's "Yellow Book"**, or program guidance document, is entering the

final phase of review and the updated document will be available later this spring. The updated "Yellow Book" will have a new look and feel that should make it easier to find any information a lab may need quickly.

It will include several new sections on reference sample requirements and how to complete an application for certification properly. All currently certified, registered and

reciprocity labs will receive a free copy of this booklet. Other

(Continued on p. 12)

## **NEWS BRIEFS**

(Continued from p. 11)

interested parties may obtain copies of the document from Carol Lochner (608) 267-7633.

### **2) The latest issue of the LUST Release News extended the holding time for DRO**

analysis to allow solvent addition up to ten days from the date of sample collection. The September 1995 methods required solvent addition within 72 hours, but results of a recently published study demonstrated that DRO is stable for up to ten days. The Laboratory Certification Program will follow the Department's recommendation and not enforce the 72 hour holding time but the 10 day holding time instead. Questions should be addressed to Alfredo Sotomayor at (608) 266-9257.

### **3) Standards for Diaminotrazine**

and the other two regulated atrazine metabolites (desethylatrazine and deisopropylatrazine) are available from the Ciba Geigy Agriculture Division, PO Box 18300, Greensboro, N.C., 27419. However, Ciba Geigy will not release standards to laboratories without prior approval. The DNR is working with the company to obtain blanket approval for all Wisconsin certified pesticide labs, which will allow them to order standards directly from Ciba Geigy. Alternatively, laboratories may order standards for the atrazine metabolites from ERA or APG. Contact Jeff Ripp at (608) 267-0579 or Ciba Geigy's Robert Williams at (910) 632-6000 for more information on the status of the agreement.

### **4) Two Public Informational Meetings**

have been scheduled by

the Laboratory Certification Program on April 30 and May 1 regarding the recent rule changes to NR 149, 219 & 700 and other Lab Cert issues. Interested parties are encouraged to attend what should be very informal question and answer type sessions. Contact Jeff Ripp at (608) 267-0579 for more information about the meetings.

■ **April 30-** The first meeting will be held on April 30, 1996 from 11:00 - 2:00 at the State Office Building, 141 NW Barstow St. (Rm 120) in Waukesha. Take HWY 164 South (exit 295) off of I-94, go through three stoplights, turn left on Barstow St. and go two blocks to the State Office Bldg. Parking is available in the visitor spaces. There is a Hardee's Restaurant across the street, and interested parties are welcome to bring their own lunches.

■ **May 1-** The second meeting will be held on May 1, 1996 from 12:00 - 4:00 at the Portage County Courthouse, 1516 Church St., Stevens Point, WI. Off of HWY 51, take HWY 10 West to Business 51 South. Go west on Ellis St. 4 blocks. Interested parties are welcome to bring their own lunches.

### **5) What's new with Oil and Grease**

since last year? EPA proposed the HEM method with a six month period for phasing out the freon method. EPA originally hoped to promulgate the final rule by May or June but this may be delayed by the current situation with their budget. Regions 4 and 8 have granted limited alternate test procedure approvals for HEM. Region 5 is considering a similar action. Until regulations were in place, wastewater staff could accept the HEM method but could not require or recommend its use. As of March 1, 1996, the HEM procedure is officially promulgated in NR 219. In our last newsletter, we notified laboratories that the revised application fee would be waived for

this test until December 31, 1995. Few laboratories took advantage of this "free offer". Certification is required before results can be reported for compliance with a WPDES permit and some pretreatment ordinances. We encourage laboratories to apply for HEM soon so they will be able to respond favorably to requests for this method.

### **6) Reference Sample Grading Procedures**

may vary from provider to provider, however the Program would like to stress the following criteria are always used when scoring multiple-analyte samples for organics. A laboratory must pass 80% of the compounds and levels in a sample to receive acceptable results. If a laboratory fails several analytes from a particular study, and ends up with less than 80% correct, the lab must submit full results from another provider or later study in order to have acceptable results. For example, it is **not** acceptable to analyze and report those compounds which were missed in the first study. If a lab misses benzene and dichloromethane in one VOC study, they must analyze and report all of the VOCs in another study (not just benzene and dichloromethane). Also, be very careful not to report a "less than" value for any reference samples unless the sample is truly below your detection limit. At best, the data will be unusable and at worst you will receive a failure for that analyte. If your lab reports a value of "<X" and the true value is greater than X, you will receive a fail. If your lab reports "<X" and the true value is less than X, the data will be considered unusable.

### **7) The Department's internet gopher**

site has not been updated on a regular basis because the DNR has focused efforts on the World Wide Web project. We apologize to those of you who have tried to access information from our

site unsuccessfully. Hopefully, we will be able to keep up-to-date information available on the web as our computer techies learn more about managing our server. The DNR's web page is located at <http://www.dnr.state.wi.us>.

## **SECRETARY MEYER RECOGNIZES MODINE MANUFACTURING COMPANY AND TOWN OF BLOOMER LABORATORIES**

Secretary George Meyer presented Modine Manufacturing Company and the Town of Bloomer Wastewater Treatment Plant with the 1996 registered "Laboratory of the Year" awards at the March 27, 1996 Natural Resources Board Meeting. Both Modine Manufacturing Co., winner of the large registered lab

award, and the Bloomer WWTP, winner of the small registered lab award, have shown an outstanding and long term commitment to producing high quality data used as the basis for many decisions made by the DNR in the protection and conservation of Wisconsin's natural resources. Congratulations to both

laboratories and thanks for a job well done! Nominations for next year's awards are due prior to December 31, 1996. Contact Debra Piper at

(608) 267-7633 for more information. Program files are always open to the public. We encourage anybody wishing to stop by the Central Office to make prior arrangements with Carol Lochner at (608) 267-7633.

✓ If you have questions about the program or would like to request information, applications or copies of a report call the program's general phone number (608) 267-7633. We also have a dedicated fax line for all correspondence (608) 267-5231.

✓ The DNR's World Wide Web Page is now up and active, however we are still working on the Laboratory Certification Home Page. This issue of LabNotes is published on the web. The address is <http://www.dnr.state.wi.us>.

Facility Identification Number on all correspondence with the program. This helps tremendously for filing.

**Clarification-** The Department would like to clarify when confirmation is required for volatiles on a LUST site (NR 700 series) and who is responsible for seeing that it is done. Strictly, confirmation is an option available to responsible parties (RP's). If the RP (or the consultant representing them) intends to question the validity of low-level hits (i.e. detection of concentrations between the LOD and the LOQ for compounds whose soil standard is below the LOD), then confirmation is required. If they do not question the results falling within this range, then confirmation is not needed.

from the laboratory's perspective, the client (consultant) is responsible for requesting confirmation of analytical sample results.

**Application Reminder -** The laboratory certification program has revised its application for certification. The new application form is dated 1/1/96 and is distinctly different from previous versions of the form. Please discard any older application forms because they are outdated and the fees are incorrect. Also, laboratories applying after June 30, 1996 will be required to pay the new fees (see page 2). Contact Carol Lochner at (608) 267-7633 to obtain copies of the new form.

#### LABNOTES - SPRING 1996

Wisconsin Department of Natural Resources  
P.O. Box 7921

DNR Secretary George Meyer presents the small registered "Laboratory of the Year" award to Bloomer's director of public works

DNR Secretary George Meyer presents the large registered "Laboratory of the Year" award to Modine Manufacturing representatives Robert Oertel and Jim Kinscher.

